



Fractional Thermal Runaway Calorimetry (FTRC)

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Representing the Fractional Thermal Runaway Calorimeter Development Teams



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Small-format (S-FTRC) and Large-format (L-FTRC) Fractional Thermal Runaway Calorimeter Development Teams

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Advantages of Lithium-Ion Cells

Advances in battery technology have led to ever-increasing specific energy (W-hr/kg) and specific power (W/kg).

Today's secondary (rechargeable) lithium-ion batteries have demonstrated specific energies in the 100-200 W-hr/kg range with demonstrated specific powers as high as 500 W/kg.

Older aerospace systems used battery chemistries such as nickel-cadmium with about four times less specific power and nickel-hydrogen with about two times less specific power. These batteries also had thermal challenges when they were first introduced but the thermal challenges with lithium-ion cells are even more severe due to the higher energy density.

The excellent, mass-efficient energy storage capability of lithium-ion batteries has led to their use on many aerospace platforms and on countless diverse commercial products including laptops, electric cars and more recently hover boards.

How Does a Lithium-Ion Cell Work?

A battery is made up of an anode, cathode, separator, electrolyte, and two current collectors.

Electrolyte carries positively charged Lithium-ions from the anode to the cathode and vice versa through the separator.

Movement of the Lithium-ions creates free electrons in the anode which creates a charge at the positive current collector.

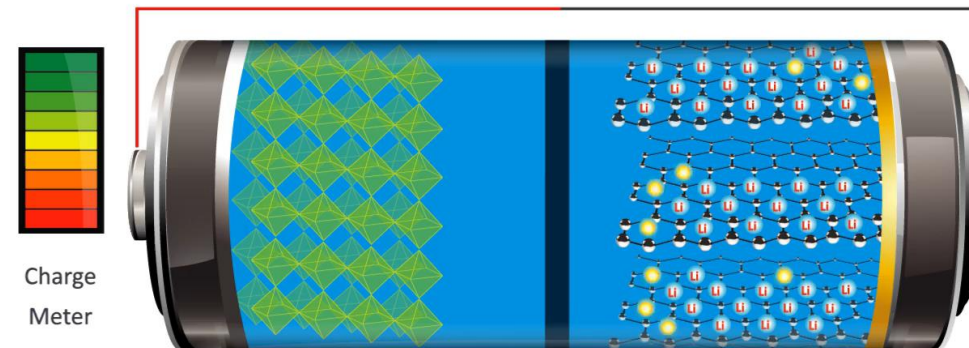
Electrical current flows from the current collector through a device and to the negative current collector. The separator blocks the flow of electrons inside the battery.

During discharge, the anode releases Lithium-ions to the cathode, generating a flow of electrons from one side to the other.

During charging, the opposite happens: Lithium-ions are released by the cathode and received by the anode.

How Lithium-ion Batteries Work

Discharge



Lithium-Ion Cell Chemistries

There are a number of Lithium-Ion cell chemistries including:

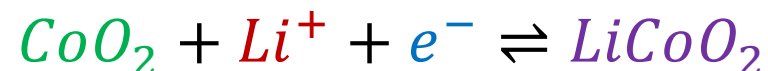
Name	Chemical Symbol	Also Known As
Lithium iron phosphate	LiFePO_4	IFR or LFP or Li-phosphate
Lithium manganese oxide	LiMn_2O_4	IMR or LMO or Li-manganese
Lithium manganese nickel	LiNiMnCoO_2	INR or NMC
Lithium nickel cobalt aluminum oxide	LiNiCoAlO_2	NCA or Li-aluminum
Lithium nickel cobalt oxide	LiNiCoO_2	NCO
Lithium cobalt oxide	LiCoO_2	ICR LCO Li-cobalt

Cell chemistries tested and reported in this presentation:

18650 Cells	Chemistry
LG 18650-HG2	INR
Samsung 18650-30Q	INR/NCA
LG 18650-MJ1	NMC
Sony 18650-VC7	NCA
Large Format Cells	
GS Yuasa LSE 134	LiCoO_2

Electrochemistry of a Lithium-Ion Cell

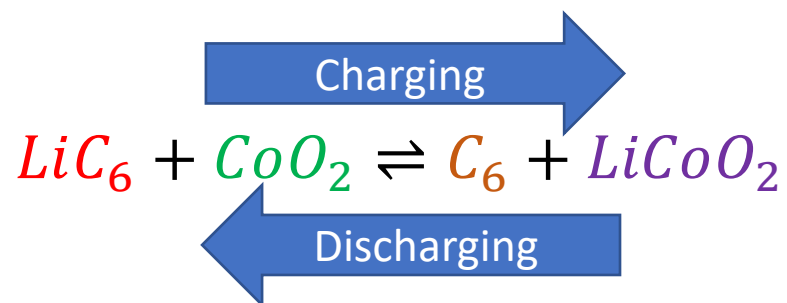
Reactions take place at both the cathode (positive electrode) and the anode (negative electrode). At the cathode:



and the anode:



The full reaction is:





Lithium-Ion Cells and Thermal Runaway

Many recent events have been reported where lithium-ion cells have rapidly released their stored energy leading to fires.

This phenomenon is referred to as thermal runaway (TR) and may arise due to thermal failure, mechanical failure, electrochemical abuse, internal short circuiting, and external short circuiting.

While TR of a single cell in a system's battery pack remains a significant event that needs to be accounted for in the system design, such an event can be more readily contained without significant ramifications.

When TR of a single cell triggers the subsequent TR of an adjacent cell or cells, it leads to a propagating event that has even more serious system impacts.

Lithium-Ion Cells and Thermal Runaway

The basic scenario for a propagating event is:

- TR of a given cell in a battery pack leads to extremely fast warming of its internal cell windings followed closely by the cell's case.
- Heat generation warms the case of a neighboring cell which warms its winding.
- When the neighbor's winding warms sufficiently, the plastic material separating the anode and cathode layers in the cell will melt allowing the anode and cathode to come into contact leading to a short within this adjacent cell and leads to the TR of the neighboring cell.
- ...and so on.

Lithium-Ion Cells and Thermal Runaway

For TR, the sources of energy include not only the stored electrical energy but also the exothermic energy released in the various chemical reactions triggered by the high temperatures including combustion within the cell fed by oxygen released from the cathode materials.

As the cell temperature increases, the vapor pressure of the electrolyte within the cell increases. When the internal pressure exceeds the maximum vent port pressure, gases and liquids vent and carry energy away from the cell which can impinge upon neighboring cells.

The TR cell presents an external short to any parallel cells, whose warming increases their propensity for TR propagation. All of these effects, and more, must be considered to simulate accurately the effects of a thermal runaway and the likelihood of propagation to an adjacent cell.

Heat Generation During Thermal Runaway Chemical Reactions

Thermal runaway involves exothermic processes and is separated into:

- electrolyte breakdown
- solid electrolyte interphase (SEI) decomposition
- cathode oxygen release
- electrolyte oxidation, and
- electrolyte oxidation in the surrounding air

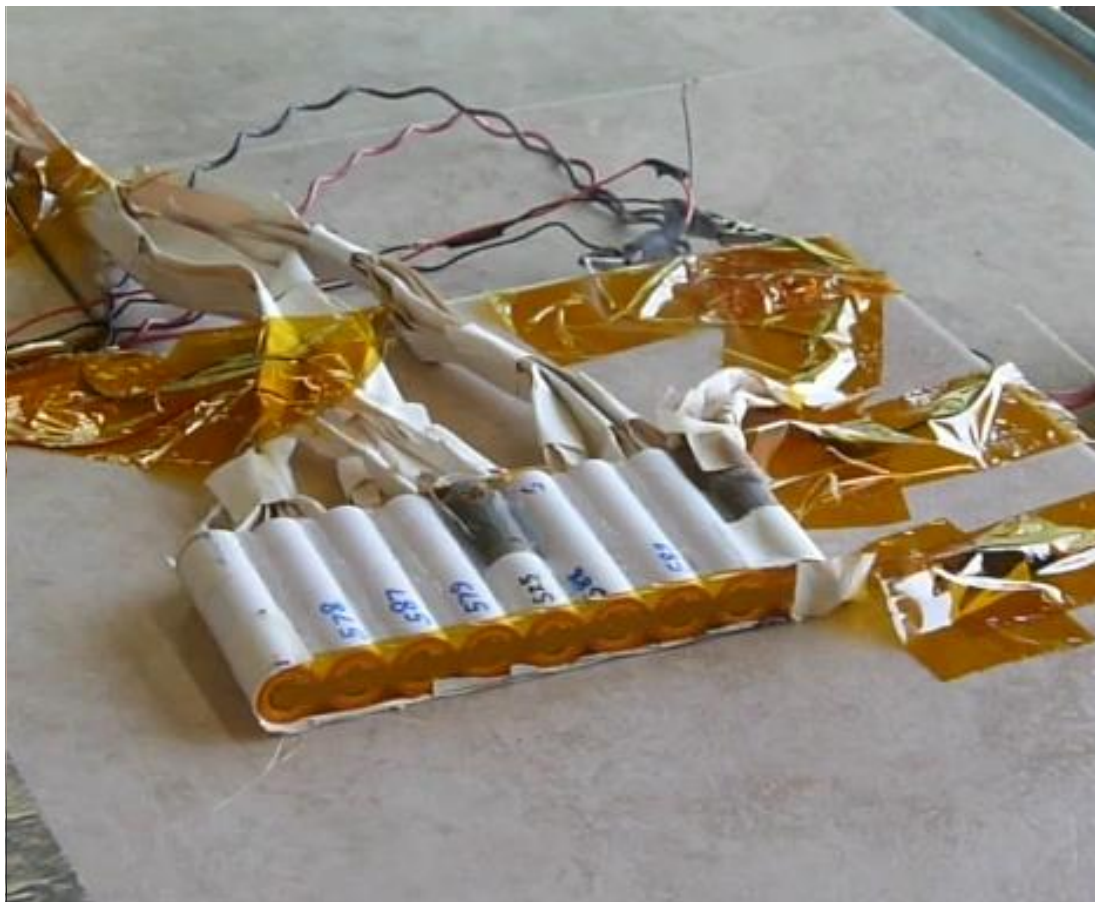
The reactions described do not include the involvement of plastics within the battery but the contribution of plastic oxidation has been estimated to be as great as the contribution of electrolyte in terms of heat release -- on the order of 2.5 MJ for plastics versus 1.92 MJ for electrolyte per kilogram of battery.

From:

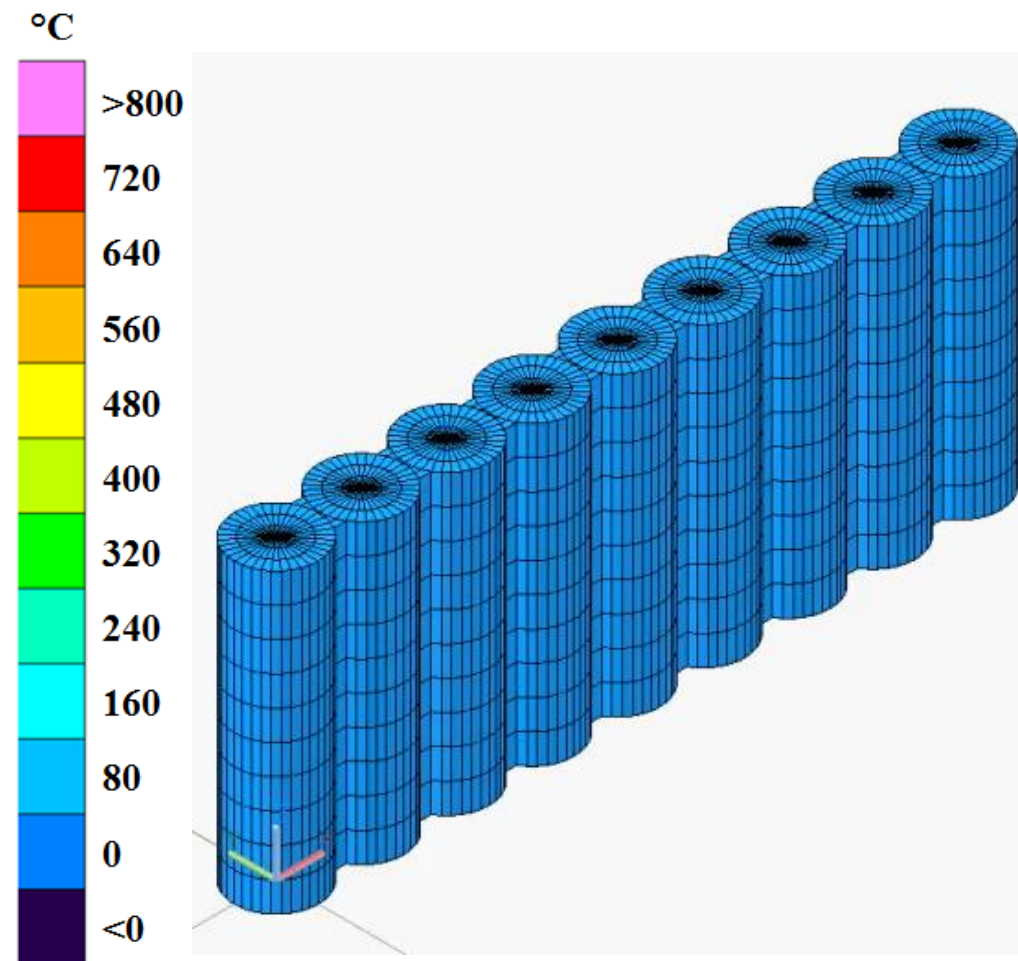
Hewson, J. C., Domino, S. P, *Thermal runaway of lithium-ion batteries and hazards of abnormal thermal environments*, SAND2015-3080C, 9th U.S. National Combustion Meeting, Cincinnati, Ohio, May 2015.

P. Ribiere, S. Grugeon, M. Morcrette, S. Boyanov, S. Laruelle, and G. Marlair. *Energy and Environ. Sci.*, 5 (2012) 5271–5280.

Thermal Runaway Propagation



Lithium-Ion Cell "Picket Fence" Test Configuration

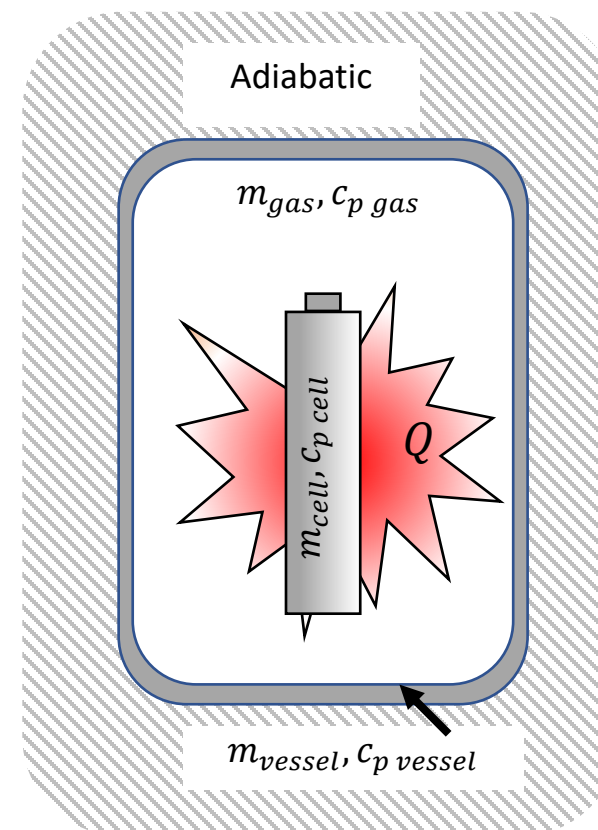


Thermal Analysis Model

What is a Calorimeter and How Does it Work?

A calorimeter is “an object used for calorimetry, or the process of measuring the heat of chemical reactions or physical changes.”*

Heat is energy liberated or generated during a process – in this case, it is the heat generated when a lithium-ion cell goes into thermal runaway.



“Ideal” Calorimeter

$$Q = m_{cell}c_{p\ cell}(T_{final\ cell} - T_0) + m_{vessel}c_{p\ vessel}(T_{vessel} - T_0) + m_{gas}c_{p\ gas}(T_{gas} - T_0)$$

Ideal Energy Balance



Small format – Fractional Thermal Runaway Calorimeter (S-FTRC)



Motivation for a New Calorimeter

Following the 2013 Boeing 787 Dreamliner incident, NASA teams developed new definitions for battery design success criteria:

- Always assume thermal runaway (TR) will eventually happen
- Design should ensure that TR event is not catastrophic
- Demonstrate that propagation to surrounding cells will not occur

Thermal management systems designed to mitigate the effects of thermal runaway and prevent cell-to-cell propagation should consider that no two runaway events are the same -- even for the same manufacturer and state-of-charge, there is a range of possible outcomes:

- Onset temperature, acceleration temperature, trigger temperature, trigger cell peak temperature and neighbor cell peak temperature
- Total energy released through sides and top of the cell body
- Cell failure type (e.g., side wall vs. top), system pressure increase, gases released and ejecta material



Motivation for a New Calorimeter

Optimization of Li-ion battery assemblies that satisfy the aforementioned strategies requires knowledge of:

- Total energy output range during TR for a single Li-ion cell
- Fraction of TR energy transferred through the cell casing
- Fraction of TR energy ejected through cell vent/burst paths



Motivation for a New Calorimeter

Lithium-ion batteries are becoming more widely used in a number of human-rated extravehicular activity (EVA) space applications on the International Space Station.

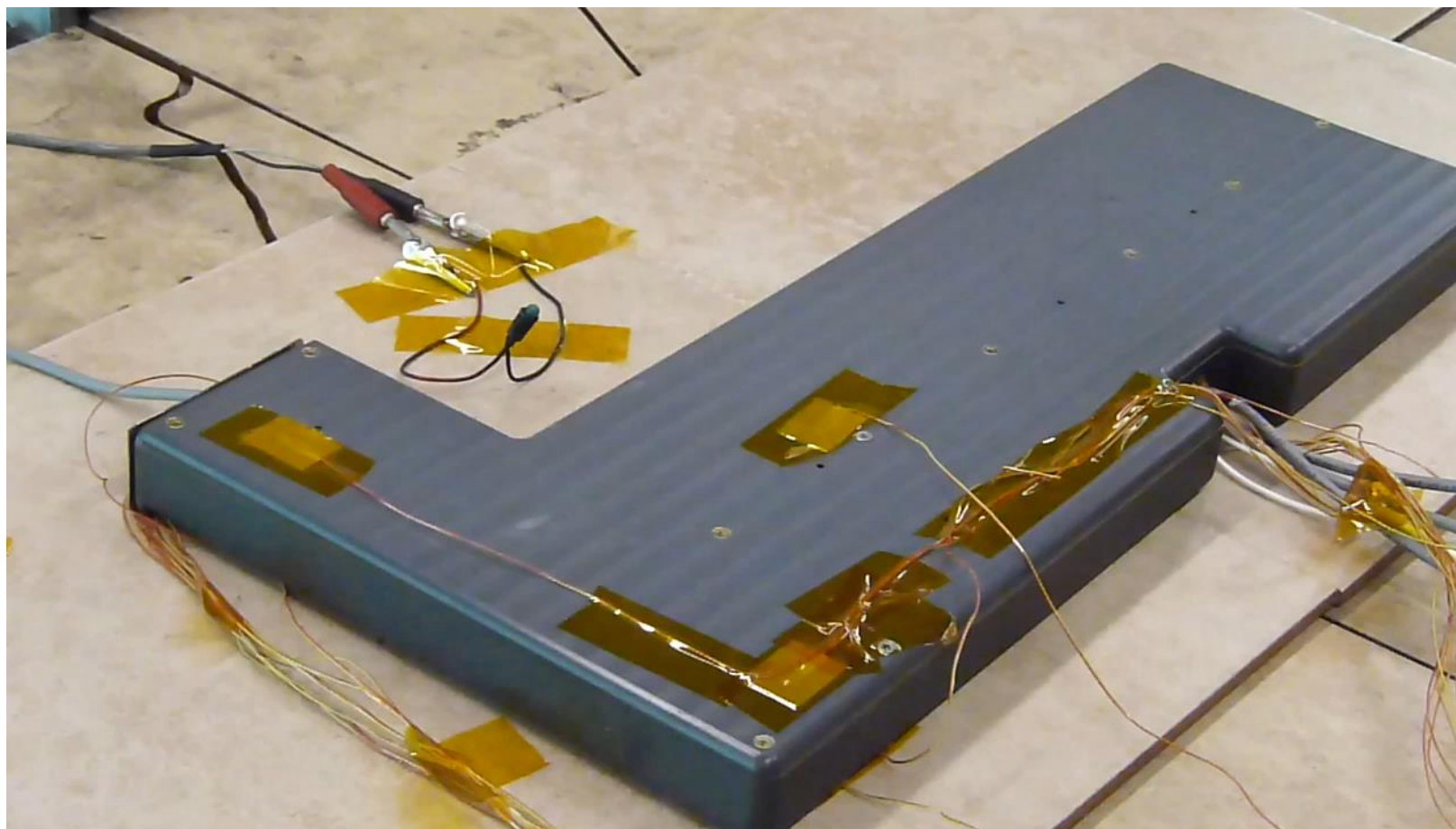
If not properly managed, testing has shown that thermal runaway in a single cell can propagate to other cells in a battery and may lead to a potentially catastrophic event.

Thermal modeling of thermal runaway propagation in the Lithium-ion Rechargeable EVA Battery Assembly (LREBA) and the Lithium-ion Pistol Grip Tool (LPGT) was pursued to inform design decisions and to understand the results of extensive development testing with the goal of enhancing safety.

A correct representation of thermal runaway in battery-level thermal models requires an understanding of internal cell triggering mechanisms, heat transfer mechanisms through the cell wall, an accounting of energy transport through vented gases and effluents and proper consideration of heat transfer mechanisms within the battery.

Motivation for a New Calorimeter

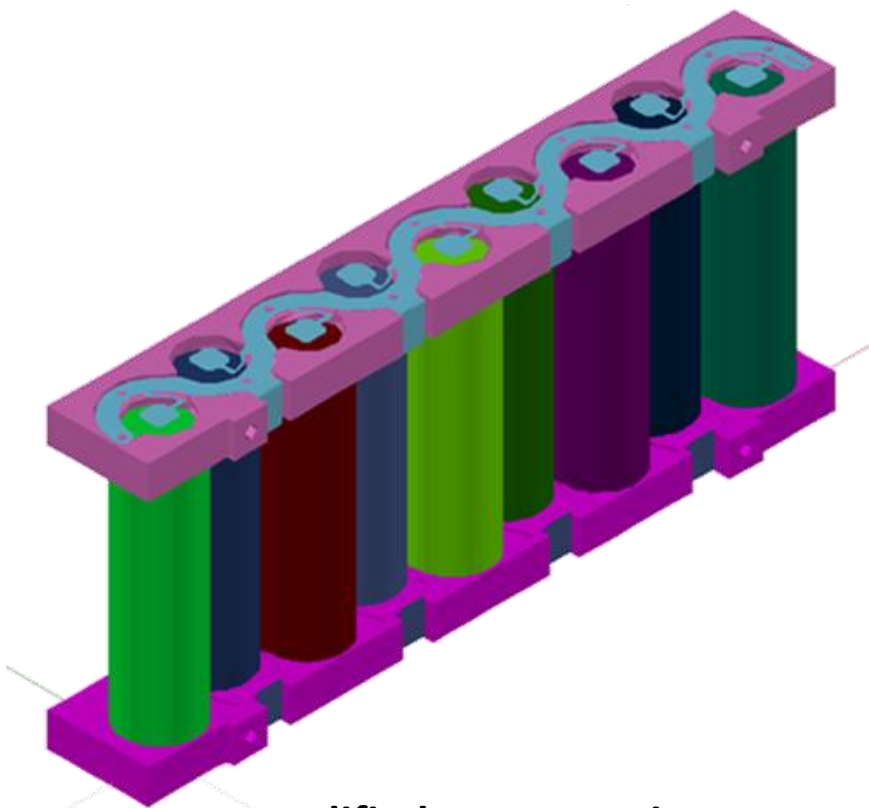
During LREBA testing, the propagation was demonstrated.



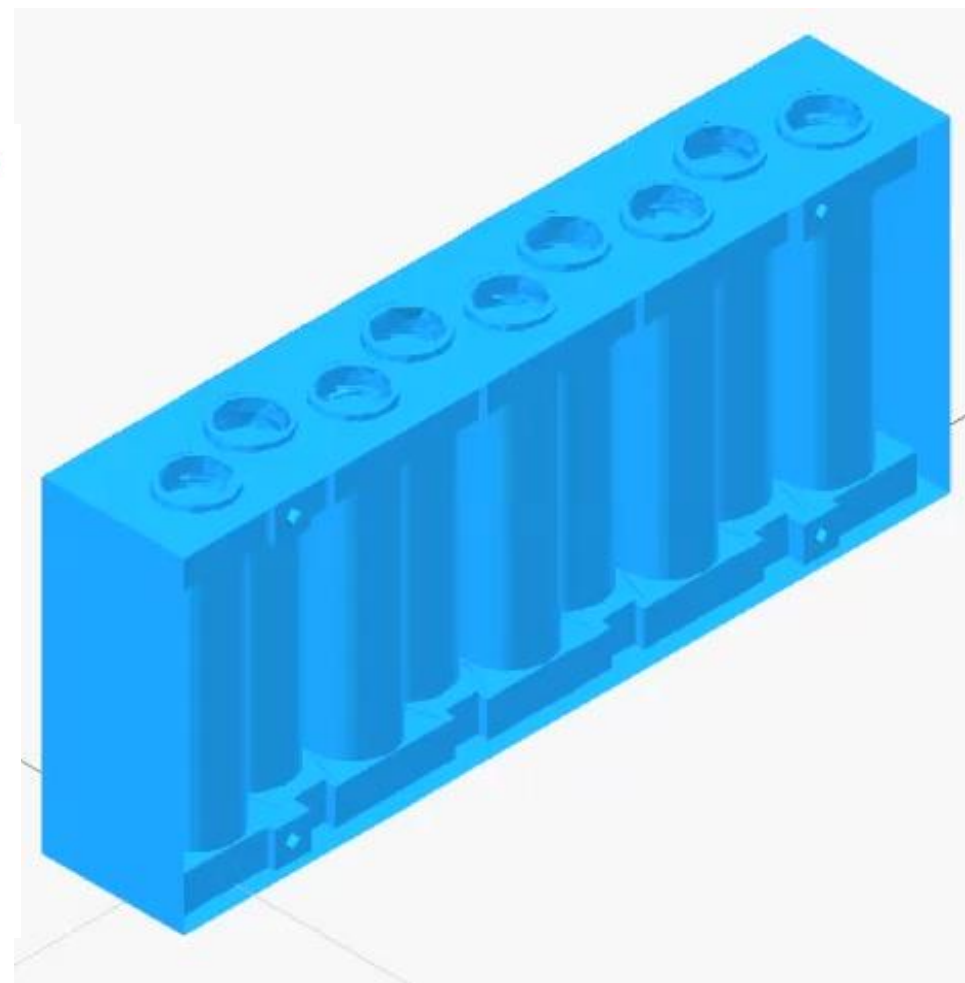
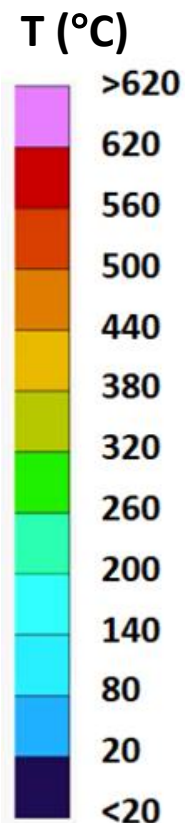
Lithium-ion Rechargeable EVA Battery Assembly (LREBA) TR Propagation Test

Motivation for a New Calorimeter

Testing and analysis led to a design change to separate the cells and provide for paths to vent gases and effluents.



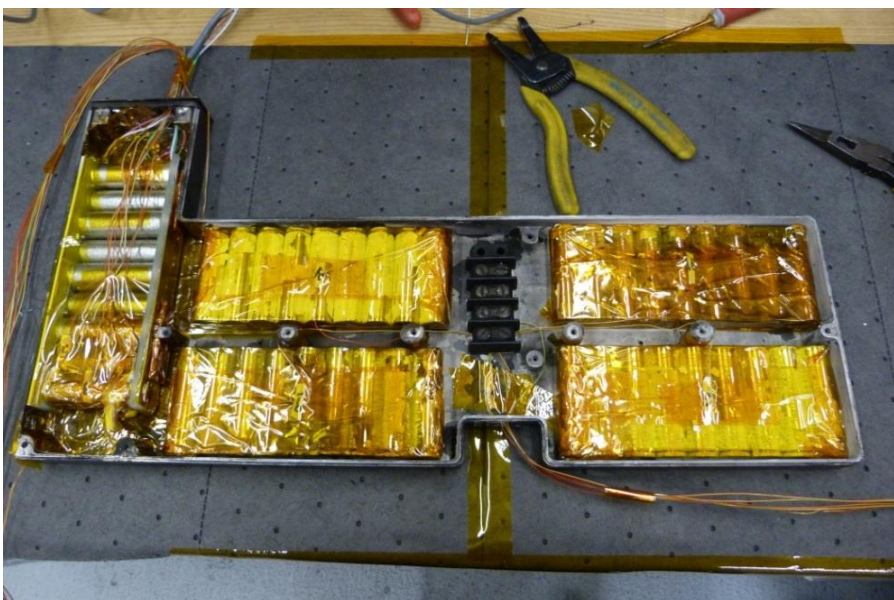
Modified LREBA Design



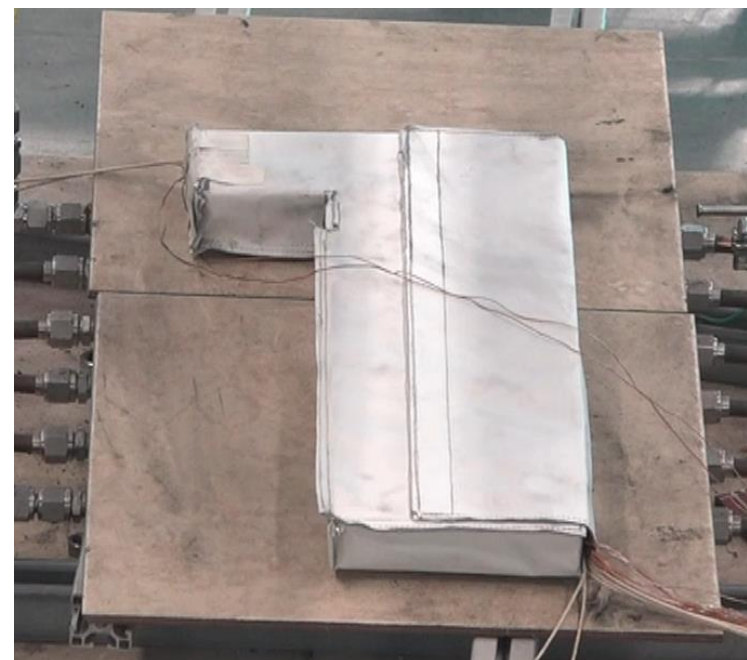
Analysis Showing TR Propagation Mitigated

Motivation for a New Calorimeter

The revised design was tested with no propagation of TR beyond the trigger cell. The work on LREBA convinced us that we needed more knowledge on total TR energy yield and how the energy was apportioned to different heat transfer modes.



**LREBA (Internal) Depicting
Redesigned 9P Cell Configuration**



**LREBA Test Showing Thermal Runaway
Does Not Propagate with the New Design**

Existing Calorimetric Techniques (1 of 2)

Accelerating Rate Calorimetry (ARC)

- Used to determine the onset of self heating/thermal runaway in cells.
- Cells are heated slowly and thermal runaway is not representative of field failures (cell venting a long time prior to the onset of thermal runaway, triggering by other means such as nail penetration).
- Due to the slow heating, turnaround time is on the order of days per test.
- While a tally of thermal energy yield is available, there is no practical means to discern thermal runaway energy fractions (via various heat transfer modes).

Bomb Calorimetry

- Adequate for tallying total heat output.
- No practical means of discerning thermal runaway energy fractions.



Existing Calorimetric Techniques (2 of 2)

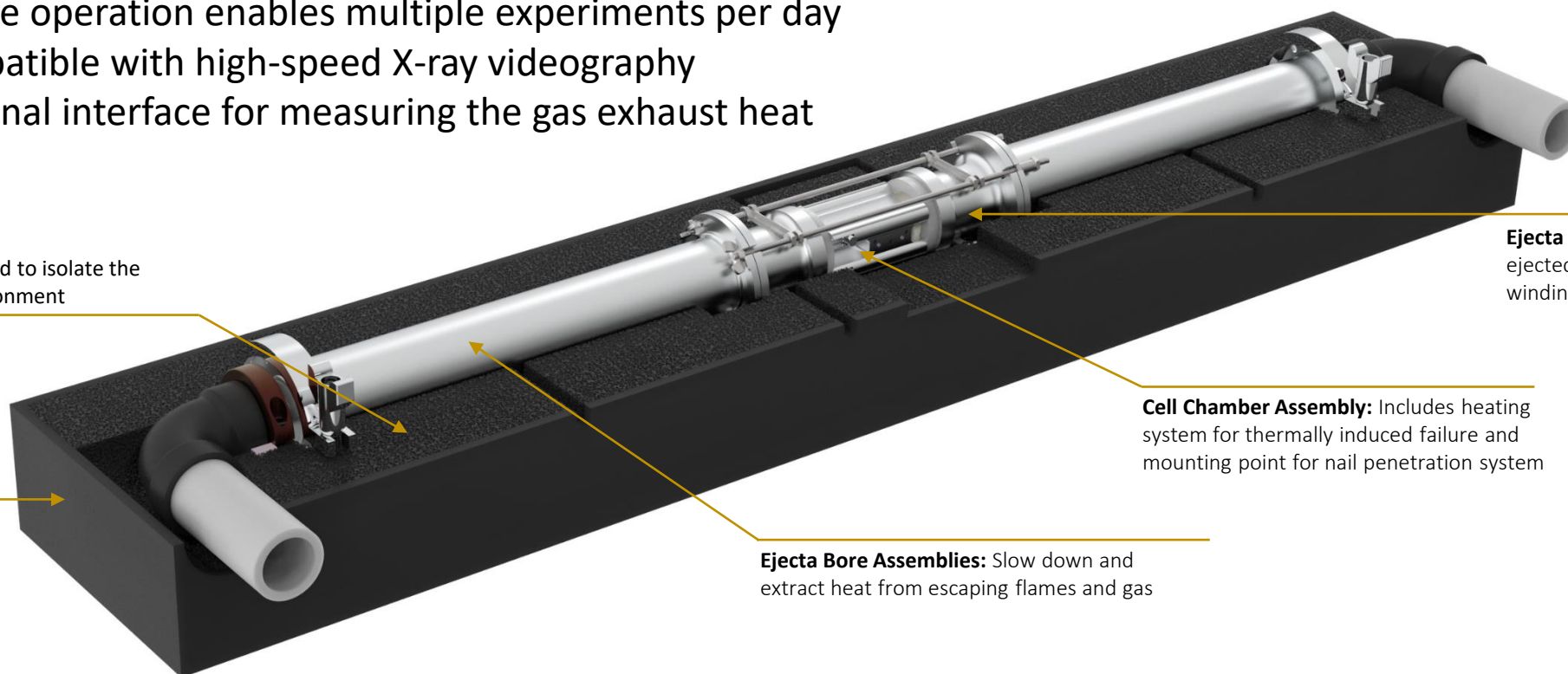
Copper Slug Calorimetry

- Effective at measuring heat output through cell casing but does not measure heat liberated in the ejecta.
- Estimates rate of mass ejection during thermal runaway.
- Must be used in conjunction with bomb calorimetry to tally ejected heat.
- No practical means of discerning thermal runaway energy fractions.

Small-format Fractional Thermal Runaway Calorimeter (S-FTRC)

NASA developed a new fractional TR calorimetry (FTRC) method for 18650-format Li-ion cells:

- Quantifies (1) total heat output and (2) fraction of heat released through the cell casing vs. ejecta material
- Energy distributions are determined by post processing temperature vs. time for each calorimeter sub-assembly
- Ambidextrous configuration accommodates cell designs with bottom vents (BVs)
- Uses high flux heaters to initiate TR quickly and is more relevant to field failure
- Simple operation enables multiple experiments per day
- Compatible with high-speed X-ray videography
- Optional interface for measuring the gas exhaust heat



Insulation: Insulation is used to isolate the calorimeter from the environment

Ejecta Mating Assemblies: Captures ejected solids such as the electrode winding

Cell Chamber Assembly: Includes heating system for thermally induced failure and mounting point for nail penetration system


Housing: Lightweight and shipping ready housing is employed to support hardware mobility

Ejecta Bore Assemblies: Slow down and extract heat from escaping flames and gas

Small-format Fractional Thermal Runaway Calorimeter (S-FTRC)

United States Patent
US 11,201,358 B1
December 14, 2021

Systems and Methods for Measuring a Heat Response of a Battery Cell in Thermal Runaway


 US011201358B1

(12) **United States Patent**
Dimpault-Darcy et al.

(10) **Patent No.:** **US 11,201,358 B1**
 (45) **Date of Patent:** **Dec. 14, 2021**

(54) **SYSTEMS AND METHODS FOR MEASURING A HEAT RESPONSE OF A BATTERY CELL IN THERMAL RUNAWAY**

(58) **Field of Classification Search**
 CPC combination set(s) only.
 See application file for complete search history.

(71) Applicant: **USA as represented by the Administrator of the National Aeronautics and Space Administration, Washington, DC (US)**

(56) **References Cited**
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 4,439,048 A 3/1984 Townsend et al.
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(72) Inventors: **Eric C Dimpault-Darcy, Houston, TX (US); John J. Darst, Houston, TX (US); William Q. Walker, Houston, TX (US); Steven L. Rickman, League City, TX (US); Natalie N. Anderson, Crosby, TX (US); Christiaan Khurana, Houston, TX (US); Bruce L. Drolen, Alhambra, CA (US); Gary Bayles, Millersville, MD (US); Zoran Bilc, Houston, TX (US)**

(73) Assignee: **United States of America as represented by the Administrator of the National Aeronautics and Space Administration, Washington, DC (US)**

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 376 days.

(21) Appl. No.: **16/175,152**

(22) Filed: **Oct. 30, 2018**

(57) **ABSTRACT**
 A system for measuring a heat response of a cell during a thermal runaway event includes a housing. An insulation is positioned within the housing. A calorimeter is positioned within the insulation and the housing. The calorimeter is configured to have the cell positioned therein. The calorimeter is configured to measure a temperature increase of the cell, one or more components of the calorimeter, or a combination thereof during the thermal runaway event of the cell. A total energy yield of the thermal runaway event is configured to be determined based at least partially upon the temperature increase. A ratio is configured to be determined based at least partially upon the temperature increase.
 (Continued)

Related U.S. Application Data

(60) Provisional application No. 62/668,596, filed on May 8, 2018.

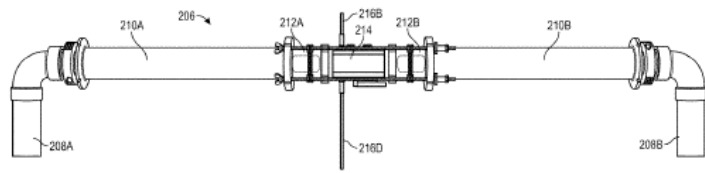
(51) **Int. Cl.**
G01K 17/00 (2006.01)
H01M 10/42 (2006.01)
H01M 10/48 (2006.01)

(52) **U.S. Cl.**
 CPC **H01M 10/4285** (2013.01); **G01K 17/00** (2013.01); **H01M 10/486** (2013.01)

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 Xuning Feng et al. "Thermal runaway features of large format prismatic lithium ion battery using extended volume accelerating rate calorimetry", Journal of Pwr Sources, vol. 255, pp. 294-301, 2014. (Year: 2014)*
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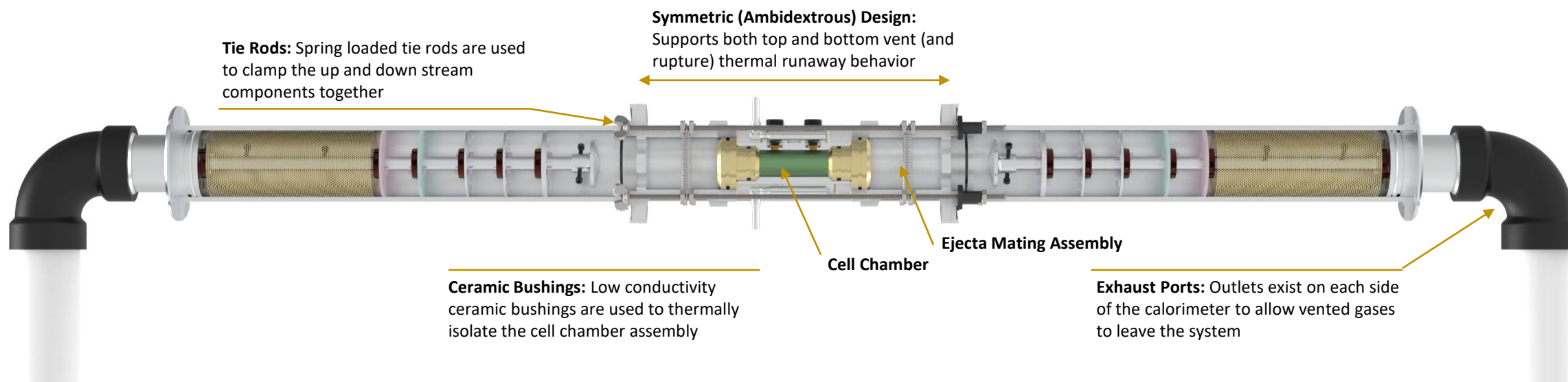
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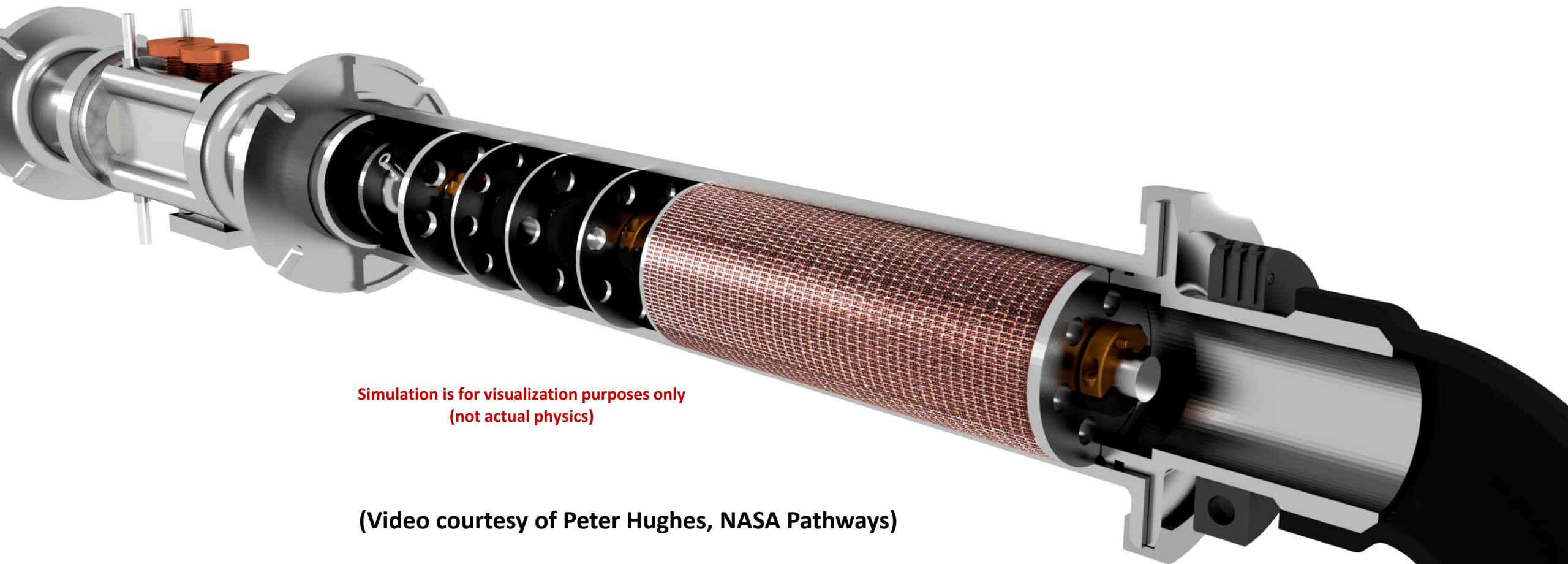
Small-format Fractional Thermal Runaway Calorimeter (S-FTRC)

- S-FTRC is designed accommodate different cell types and to characterize directional/fractional thermal runaway failure behavior (i.e., top vent, bottom vent, ruptures from any location, etc.)
- Cell chamber assembly is isolated from the remainder of the upstream and downstream calorimeter components with low thermal conductivity ceramic bushings allowing measurement of the energy fraction released through the cell casing vs. through the ejecta material
- Ejecta mating segment is designed to capture and stop complete jellyroll ejections permitting determination of the energy fraction associated with an ejected jellyroll



Small-format Fractional Thermal Runaway Calorimeter (S-FTRC)

The internal baffles and copper mesh create a tortuous path that reduces flow velocity, captures large and fine ejected particulates, and cools down the particles and gases before they exit the system.



Simulation is for visualization purposes only
(not actual physics)

(Video courtesy of Peter Hughes, NASA Pathways)

Small-format Fractional Thermal Runaway Calorimeter (S-FTRC)

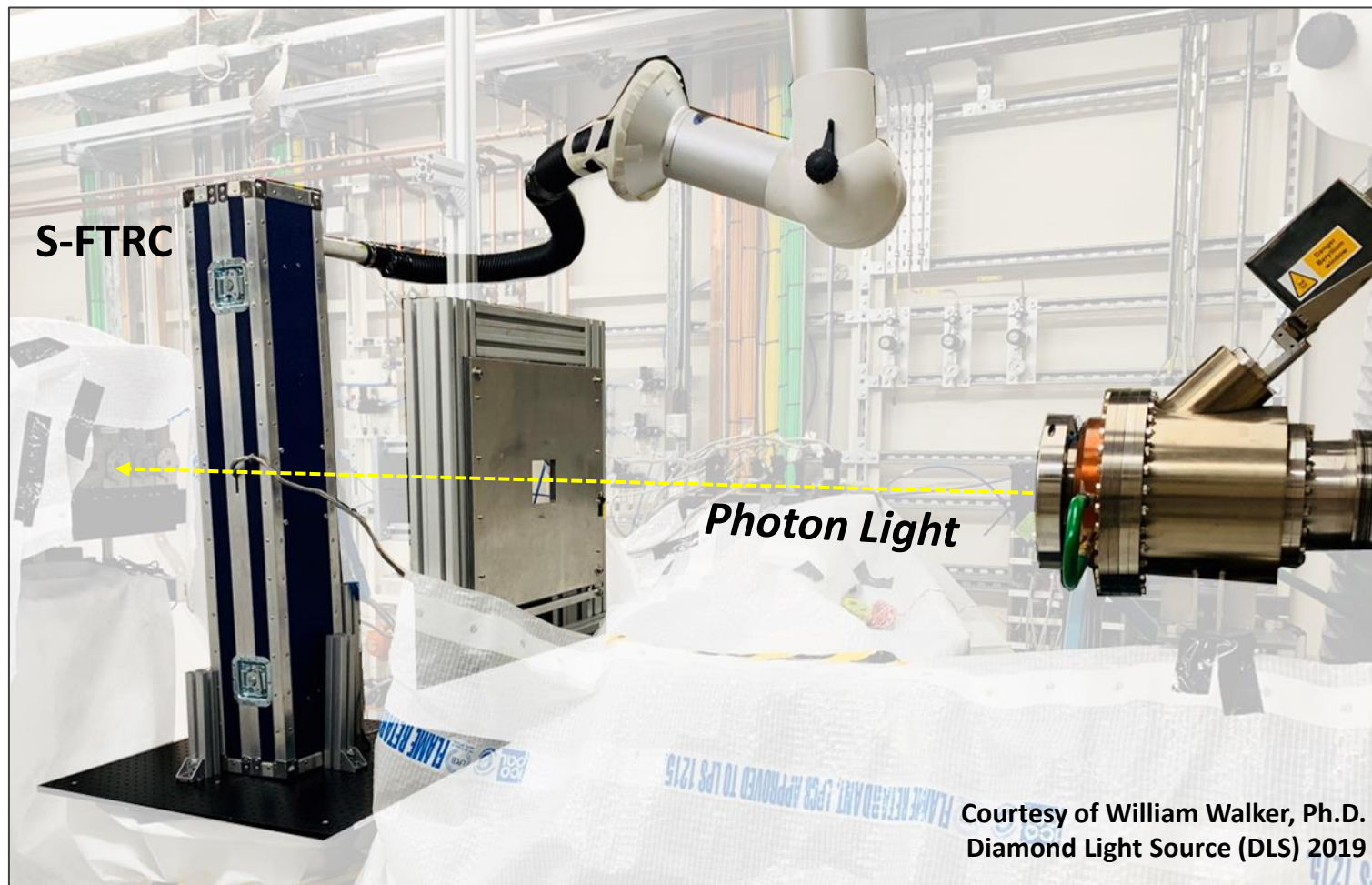


S-FTRC Test at JSC's Energy Systems Test Area

Small-format Fractional Thermal Runaway Calorimeter (S-FTRC)

S-FTRC has been used at:

- NASA JSC Energy Systems Test Area
- European Synchrotron Radiation Facility (ESRF) for in-situ high speed X-ray videography
- Diamond Light Source (DLS) Facility for in-situ high speed X-ray videography



S-FTRC Testing at Diamond Light Source (United Kingdom)

S-FTRC Results

Cell type: Li-ion 18650
Capacity: 3.5 Ah
State of Charge: 100 % (4.2 V)
Bottom vent: No
Wall thickness: Not known
Separator: Polymer
Orientation of cell: Positive end up
Location of ISCD radially: N/A
Location of ISCD longitudinally: N/A
Side of ISCD in image: N/A

Location of FOV longitudinally: Top
Frame rate: 2000 Hz
Frame dimension (Hor x Ver): 1280 x 800 pixels
Pixel size: 17.8 μm

High Speed X-Ray Videography of 18650 Cell Undergoing Thermal Runaway (courtesy of NREL/Donal Finegan, Ph. D.)

S-FTRC Results

TR results for each experiment are post-processed with primary consideration given to:

- Total TR energy yield
- Fraction of energy released through the cell casing vs. through the ejecta materials and gases
- Remaining cell mass
- Raw data derived versions of these values are referred to as the “observed” values and serve as inputs to a regression model

No two TR events are identical which results in variation of total TR energy release on a test-to-test basis:

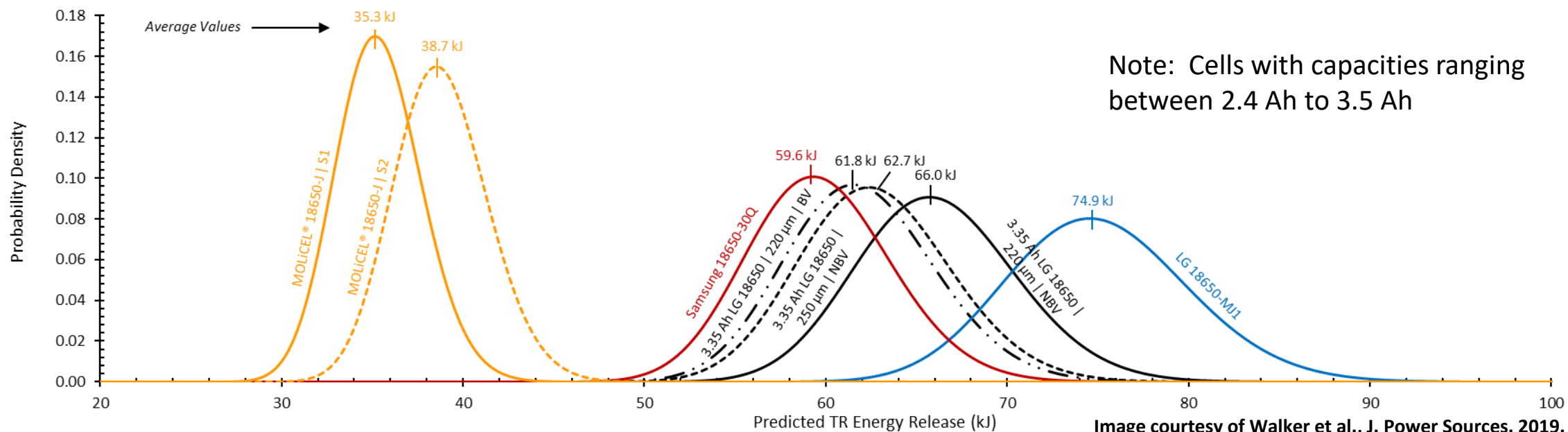
- Variation is a function of random and non-random factors associated with the experiments
- Regression model developed as a function of all S-FTRC results (e.g., cell type, failure mechanism, energy distribution, total energy release)
- Completed regression model uses the observed values for each experiment and outputs a corresponding predicted total energy release
- Predicted energy release values are used to recreate distributions to characterize the range of thermal runaway energy release

The regression model can be used to predict energy fractions as well, but at this point we are only using it to predict total energy release

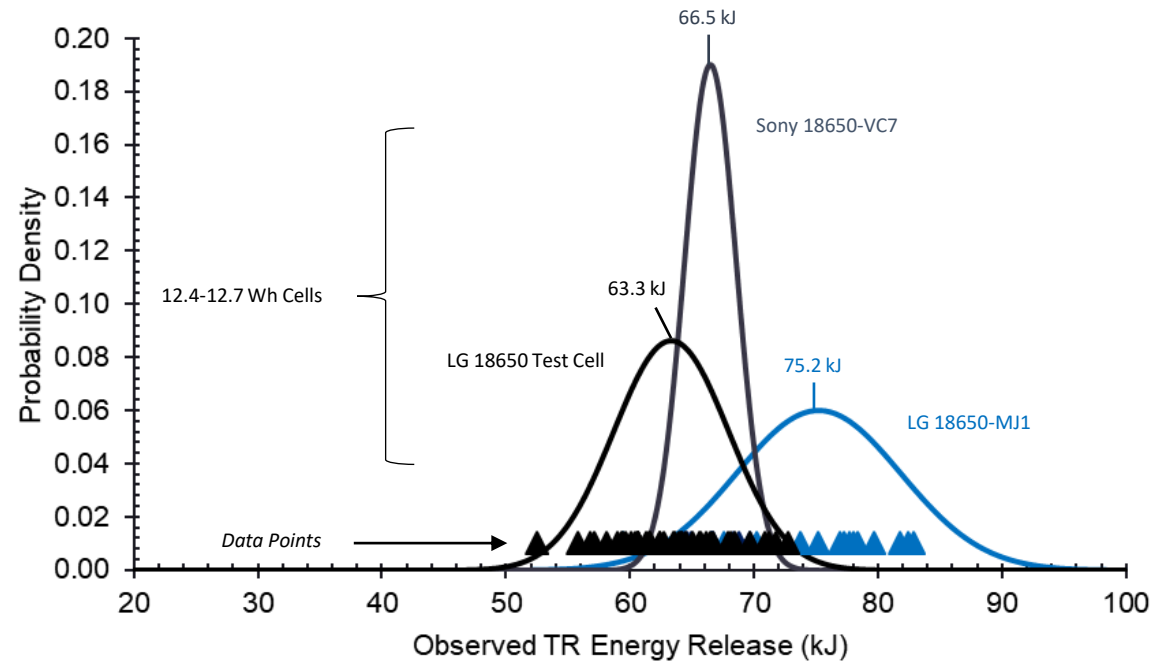
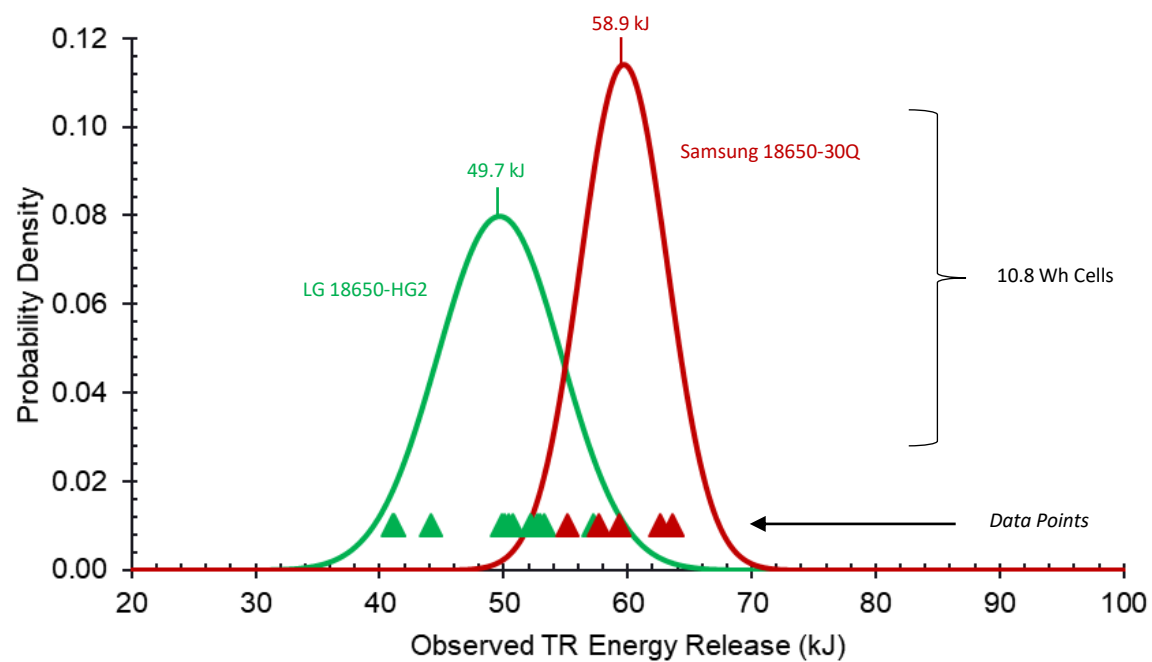
S-FTRC Results

Test-to-test variability must be taken into consideration

Regression model used to interpret and compare the S-FTRC results for total thermal runaway energy release for different cell types and varied design feature combinations (e.g., bottom vent, casing thickness, and separator material)



S-FTRC Results



S-FTRC Results

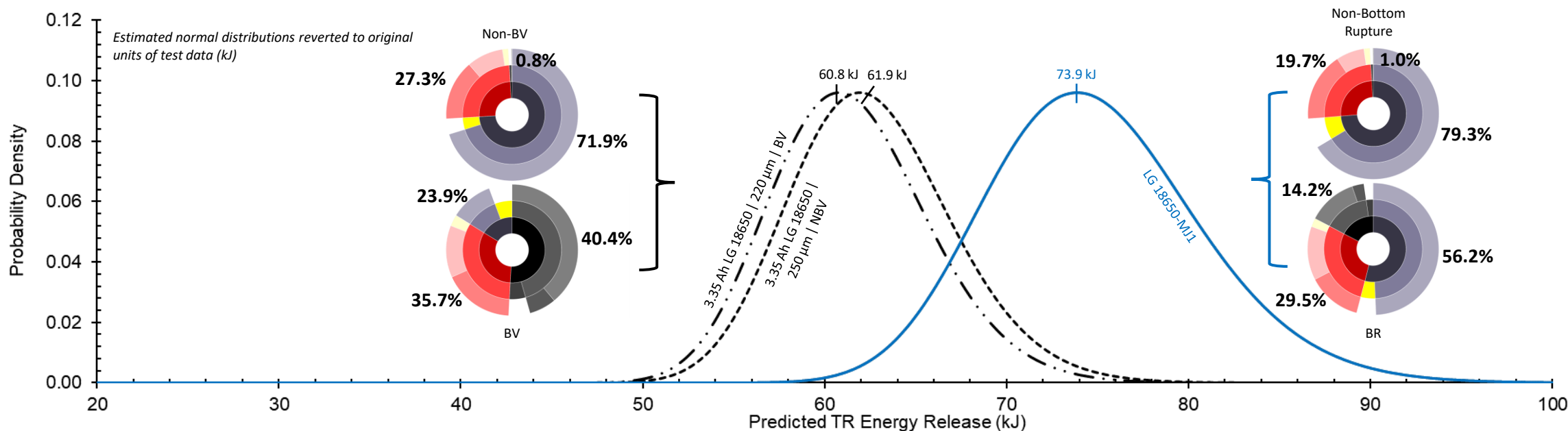
The calculated energy fractions are traceable to every calorimeter assembly, sub-assembly, and individual component



S-FTRC Results

The thermal runaway energy release fractions are determined for every cell configuration:

- Fractions can be determined from an average of all results for a given cell type or can be an average based on nominal vs. off nominal failure mechanism (e.g., top vent vs. bottom rupture)
- Fractional analysis is helpful in comparing the distribution of standard vent cells to bottom vent (BV) cells
- Standard cells typically release 20-30% of their TR energy through the cell casing and the remainder through the ejecta material



S-FTRC Results

Results provide the means to develop optimized Li-ion batteries while also maintaining safety and margin

S-FTRC, and the associated results, enables the discernment of the fractions of thermal runaway energy released through the cell casing and through the ejecta material:

- Due to the variability in thermal runaway responses, we recommend at least 10 runs to establish statistically defensible results
- Can analyze the spread of heat sources when cells rupture and compare to when they remain intact

Higher energy cells produce more heat and eject more material during thermal runaway:

- Higher magnitudes of total energy released and more violent ejections
- Less energy associated with the cell body and more energy associated with the ejecta
- The correlation is not very linear because cell enclosure design impacts the results

S-FTRC Results

Bottom vent (BV) cells released less energy (~4 kJ less for 3.35 Ah LG cell) and have higher post runaway cell mass than non-BV cells:

- BV cells produce less and more localized heat, hence a less severe and more predictable thermal runaway event as an effect of the BV feature
- Battery designers should be ready to accommodate and take advantage of cell designs with the BV feature in the future

There is not a linear relationship between stored electrochemical energy and total thermal runaway energy release

Shapiro-Wilks goodness of fit tests, quantile-quantile plotting, residual analysis, and the shape of the normal distribution curves, for all cell types, indicate that a normal distribution is an acceptable assumption



NASA and NREL Battery Failure Databank and for More Information on S-FTRC

NASA and NREL have developed an on-line Lithium-Ion cell failure databank.

More information and access to the databank may be found at:

https://www.nasa.gov/sites/default/files/atoms/files/nabw20_batt_failure_databank_walker.pdf

<https://www.nrel.gov/transportation/battery-failure.html>

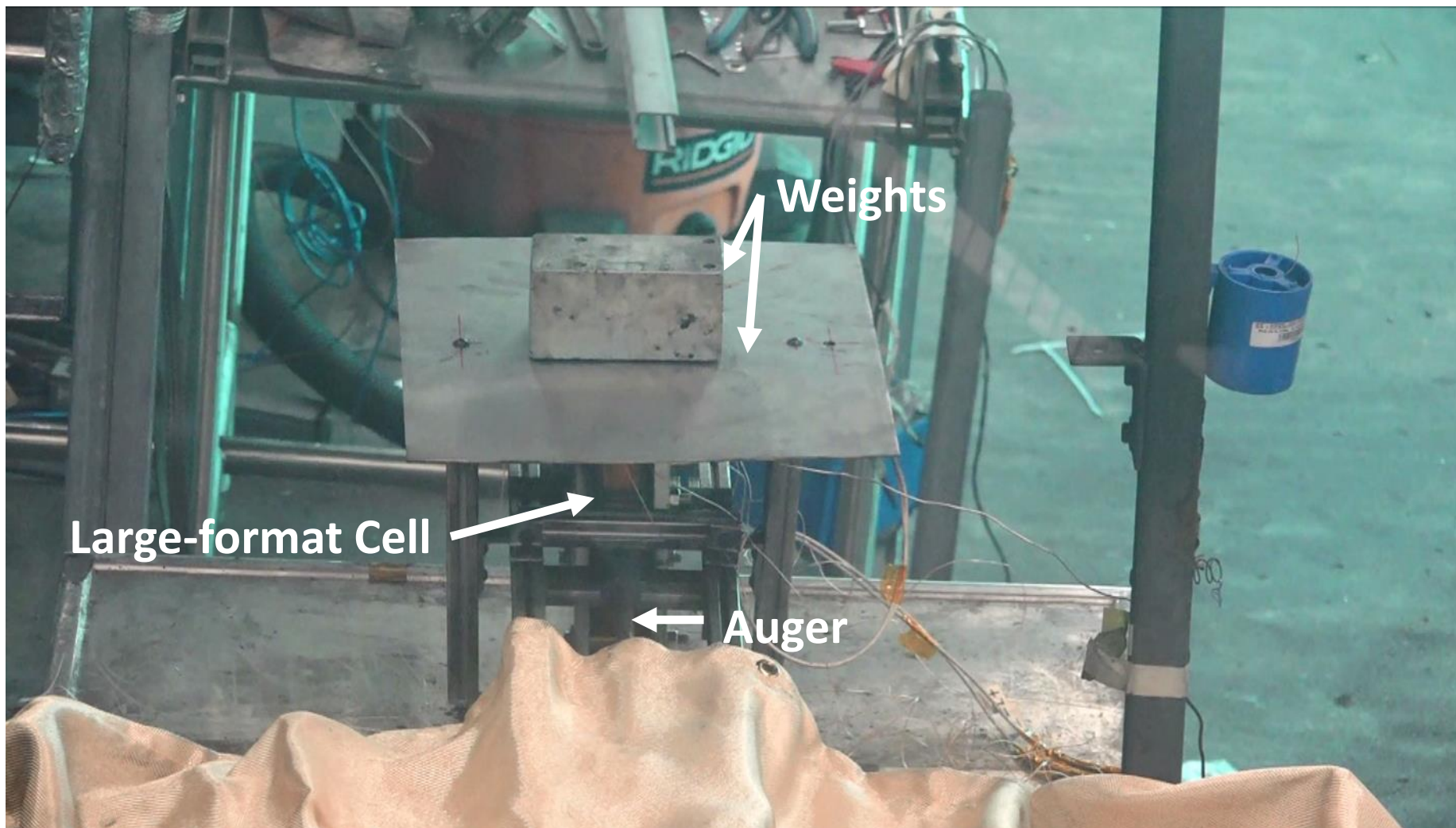
For more information on S-FTRC

<https://www.nasa.gov/ftcr>



Large format – Fractional Thermal Runaway Calorimeter (L-FTRC)

The Need for a Large Format Calorimeter



Large-format (> 100 Ah) Lithium-Ion Cell Induced into Thermal Runaway



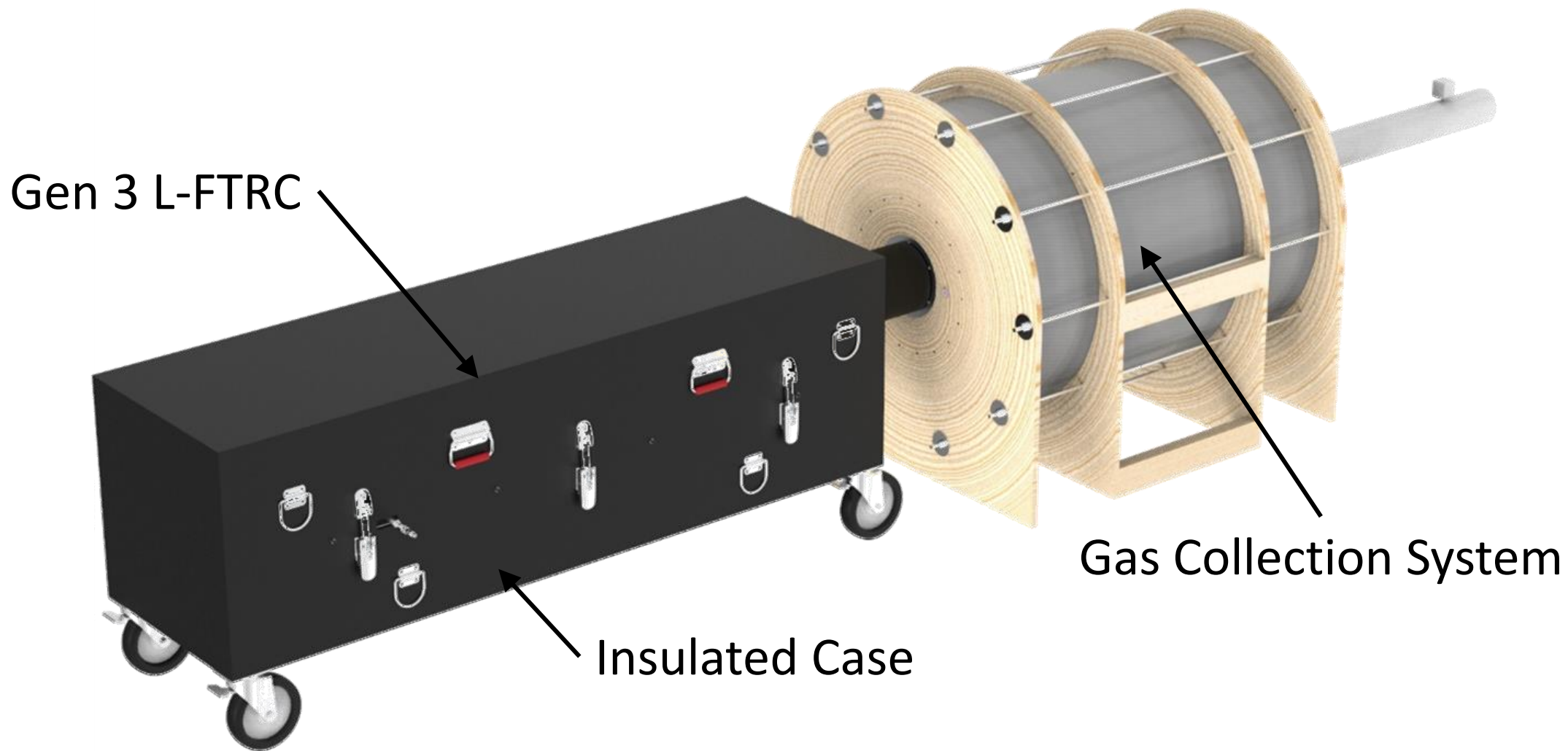
Large Format Fractional Thermal Runaway Calorimeter (L-FTRC)

A large-format Fractional Thermal Runaway Calorimeter was developed to study thermal runaway energy yield from Li-ion cells with capacities >100 Ah.

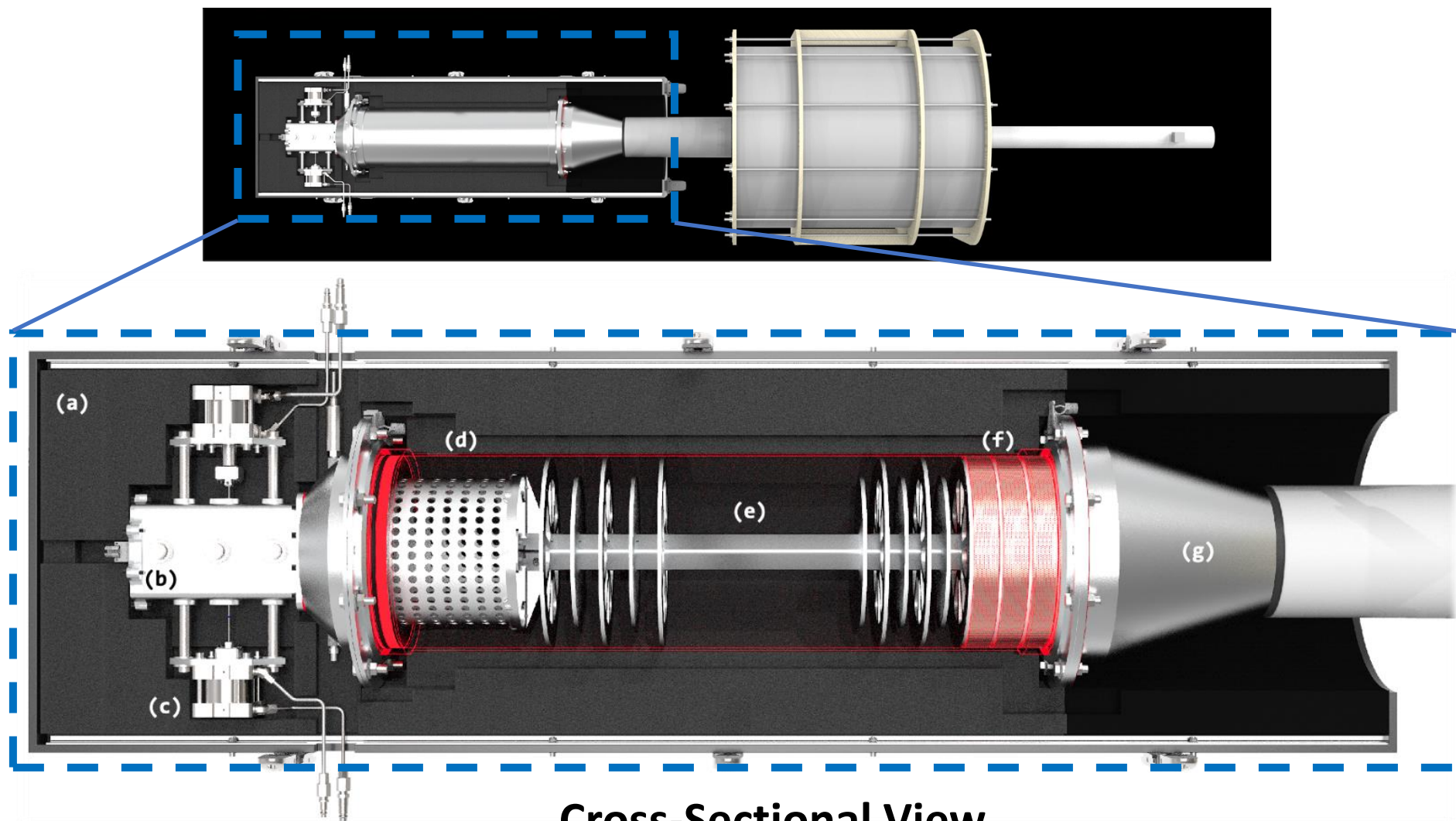
To facilitate rapid test turnaround, two hardware sets (red and green) were fabricated.

A Gas Collection System was also developed to collect and sample vent gases for analysis.

Large Format Fractional Thermal Runaway Calorimeter Configuration

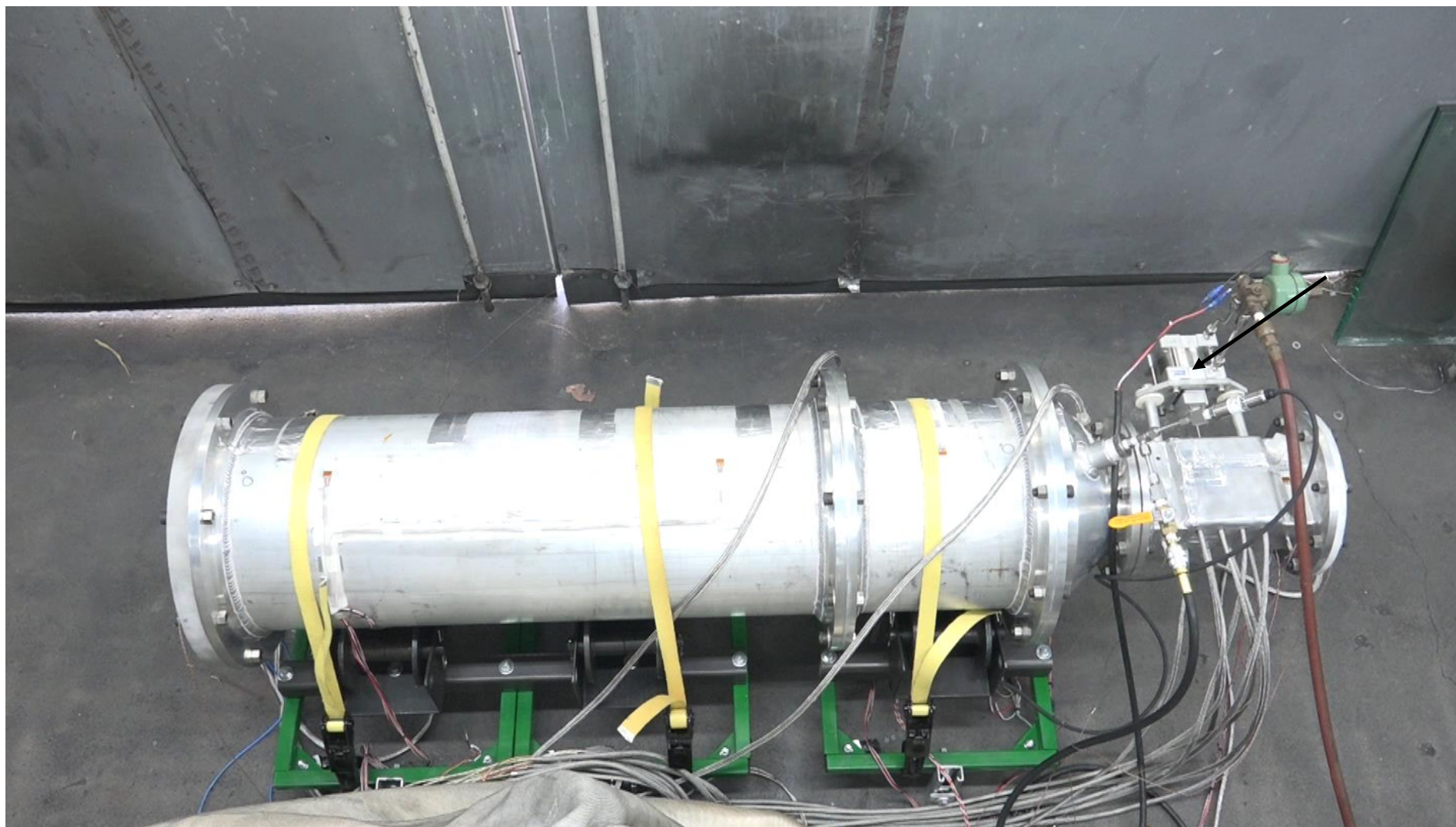


Large Format Fractional Thermal Runaway Calorimeter Configuration



Cross-Sectional View

Large Format Fractional Thermal Runaway Calorimeter Configuration



Generation 1 Calorimeter “Skeleton” Test

Large Format Fractional Thermal Runaway Calorimeter Configuration



Gas Collection System Test (End Cap Removed)



L-FTRC Test Campaign

Fourteen live-fire runs were performed using the Gen 3 L-FTRC design using 134-Ah GS Yuasa cells charged to 100% state of charge.

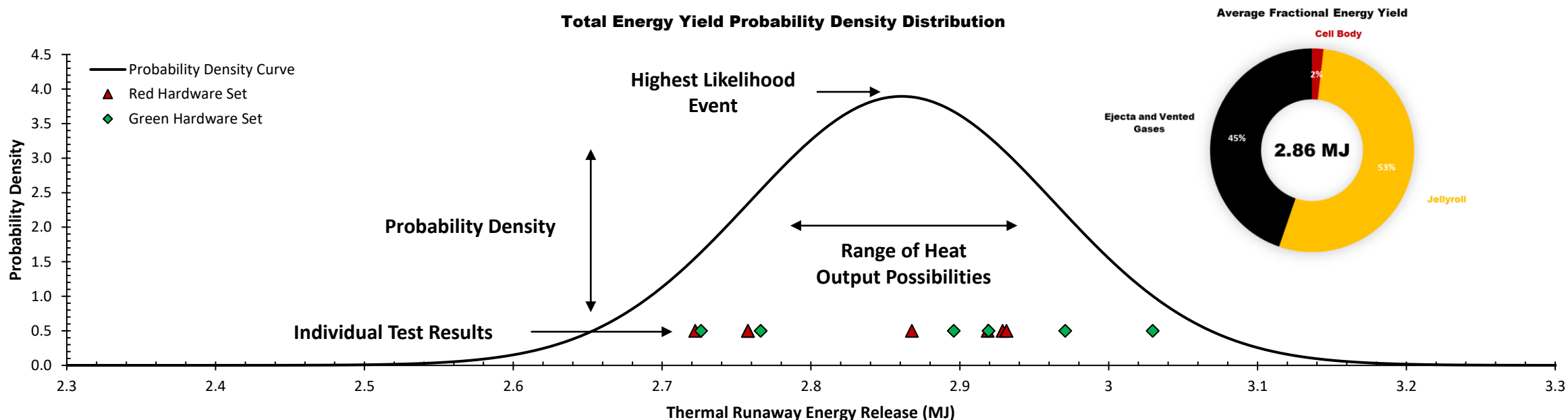
TR was induced by nail penetration (at the midpoint between the terminal and bottom end of the cell, on the narrow side) for all runs.

The Gas Collection System was used successfully for six runs.

Results are summarized on the following charts.

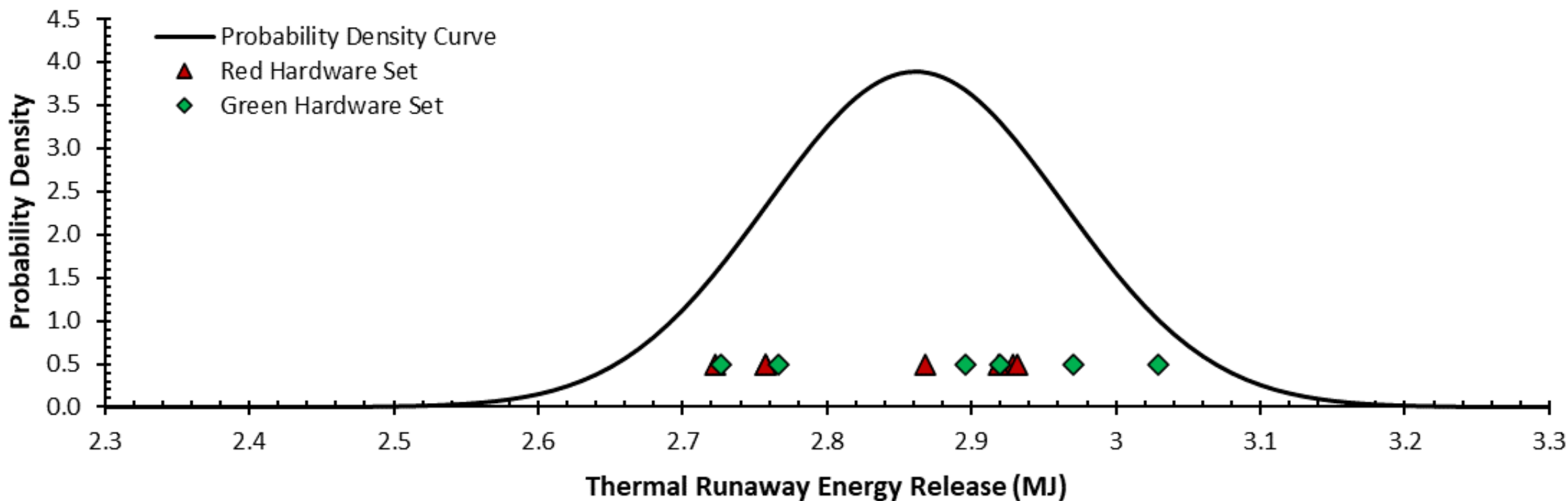
L-FTRC Results

- Test-to-test variability must be taken into consideration to characterize the overall range of expected thermal runaway behavior for a given cell type
- It is helpful to consider the variability of thermal runaway energy release as a statistical distribution to help answer the following questions:
 - What is the highest likelihood energy release? What is the lowest?
 - What is the absolute maximum energy release one could expect? Minimum?



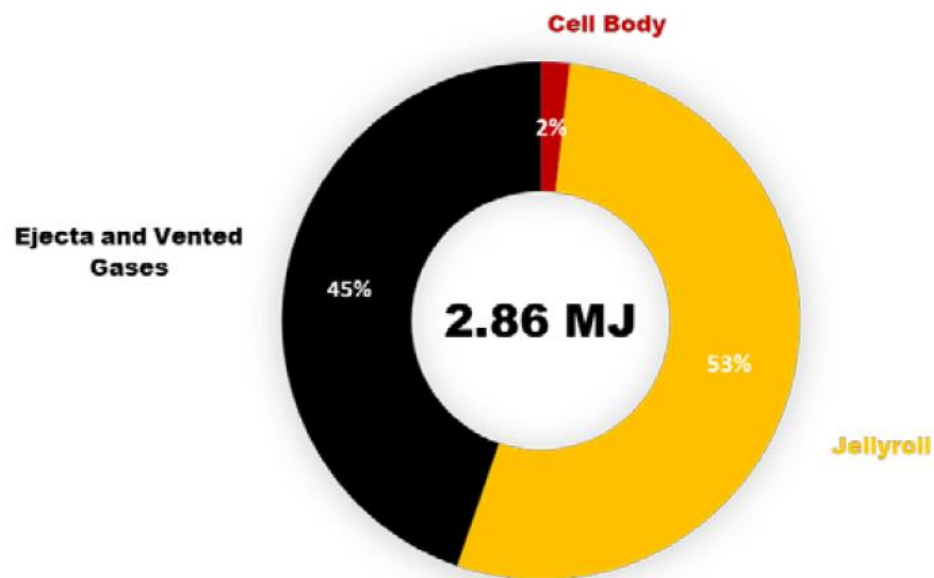
L-FTRC Results

Total Energy Yield Probability Density Distribution



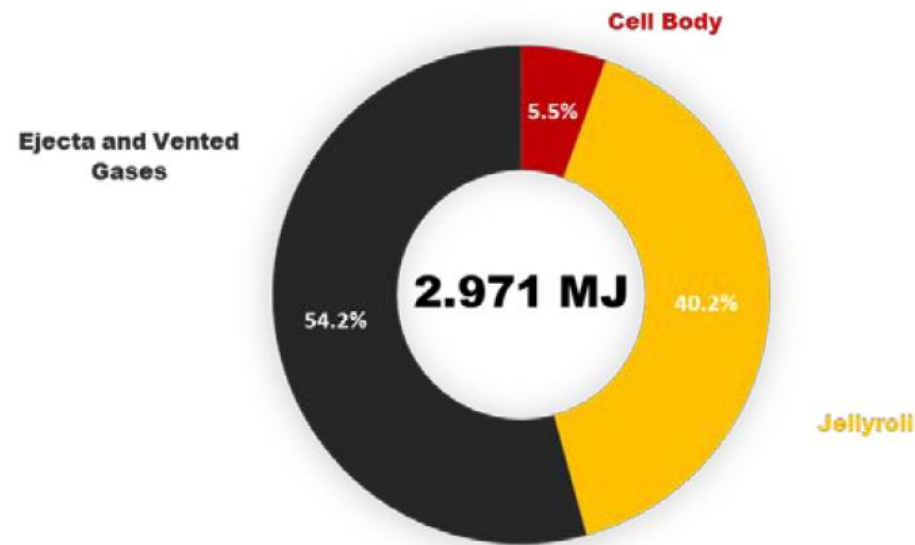
L-FTRC Results

Average Fractional Energy Yield



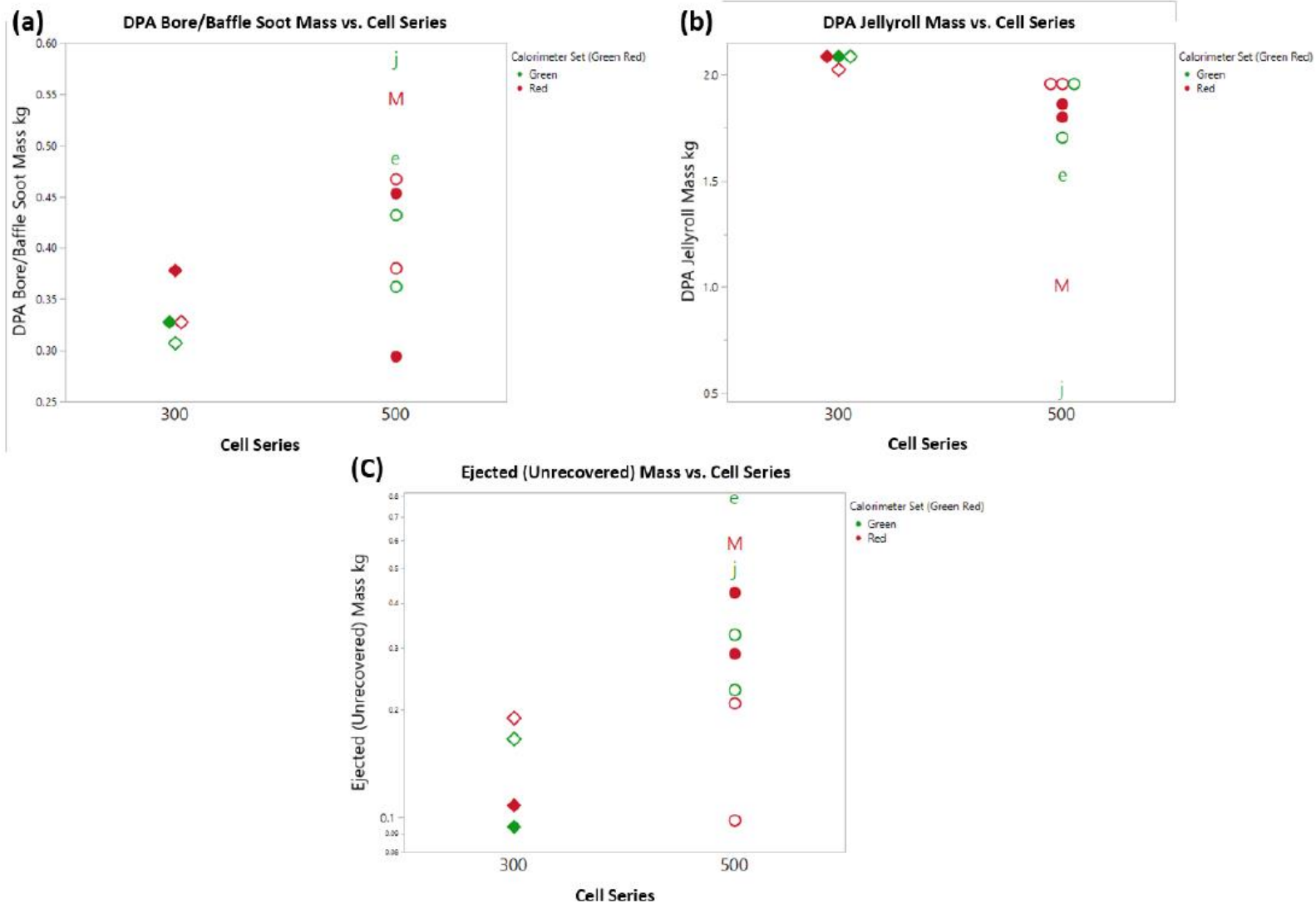
Average of Runs 2-14

Run 5 Fractional Energy Yield

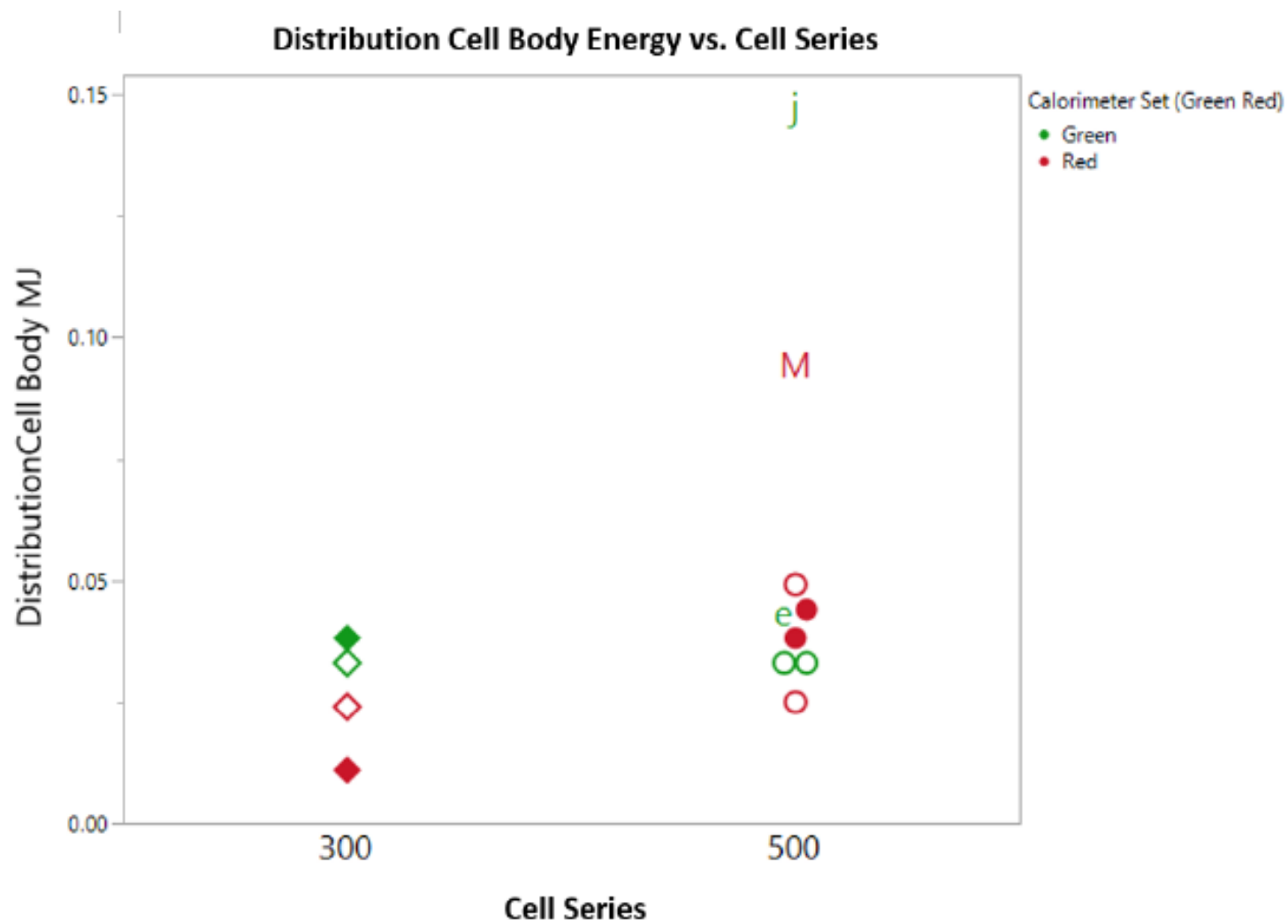


No Jelly Roll Ejection (Run 5)

L-FTRC Results



L-FTRC Results



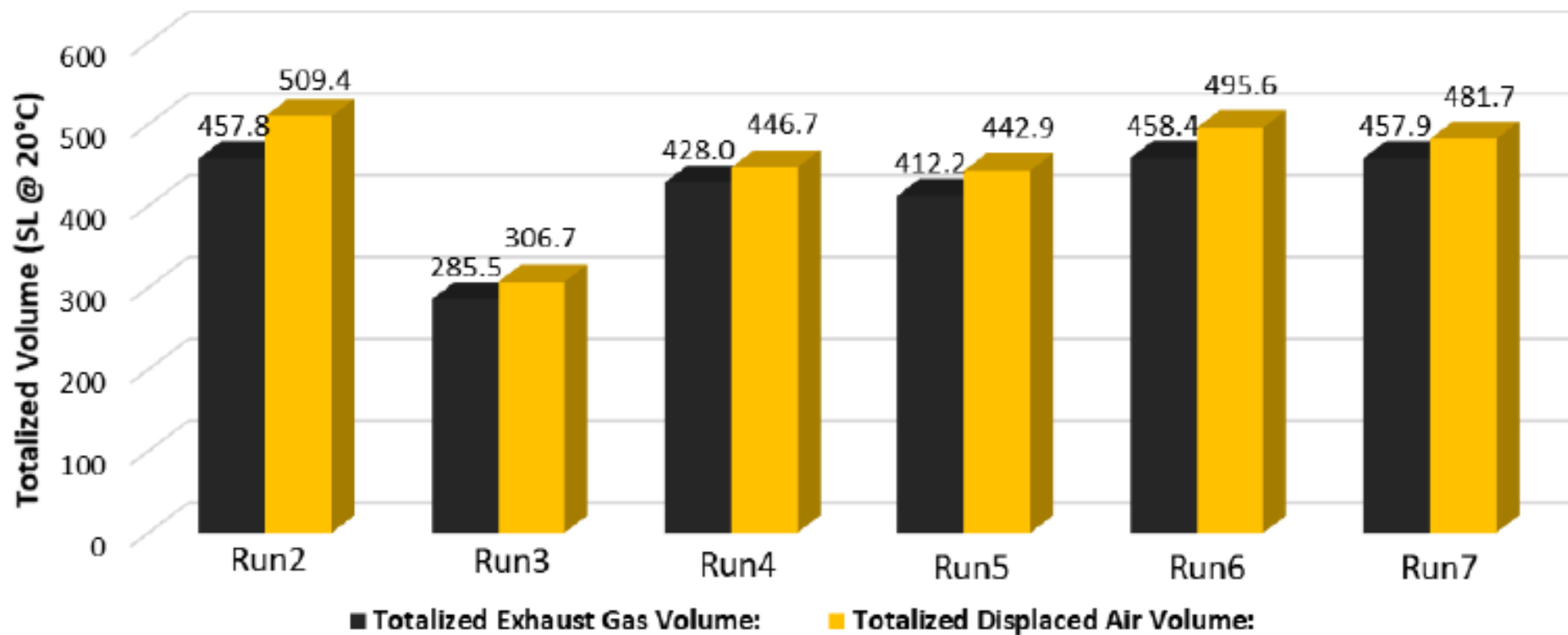
L-FTRC Results

Exhaust Gas Component	Run #1	Run #2	Run #3	Run #4	Run #5	Run #6	Run #7	Run #8
Carbon Dioxide, mole%	45%	<i>Insufficient Sample</i>	42%	42%	48%	41%	51%	52%
Hydrogen, mole%	35%		30%	35%	28%	33%	41%	35%
Oxygen, mole%	3%		2%	3%	1%	2%	3%	3%
Ethane, mole%	16%		15%	17%	15%	16%	2%	
Methane, mole%	1%		5%	4%			4%	
Additional HCs +/- 1%	< 1%		< 1%	< 1%	< 1%	< 1%	< 1%	
Dimethyl Carbonate, mole %	*	*	1.732%	*	1.457%	1.356%	*	1.618%
Ethyl Methyl Carbonate, mole %	*	*	5.096%	*	5.935%	7.149%	*	8.754%
Diethyl Carbonate, mole %	*	*	0.0249%	*	0.0323%	0.0394%	*	0.0511%
Total Mole% (Volume%)	100%		100%	100%	100%	100%	100%	100%

* No electrolyte data for these runs

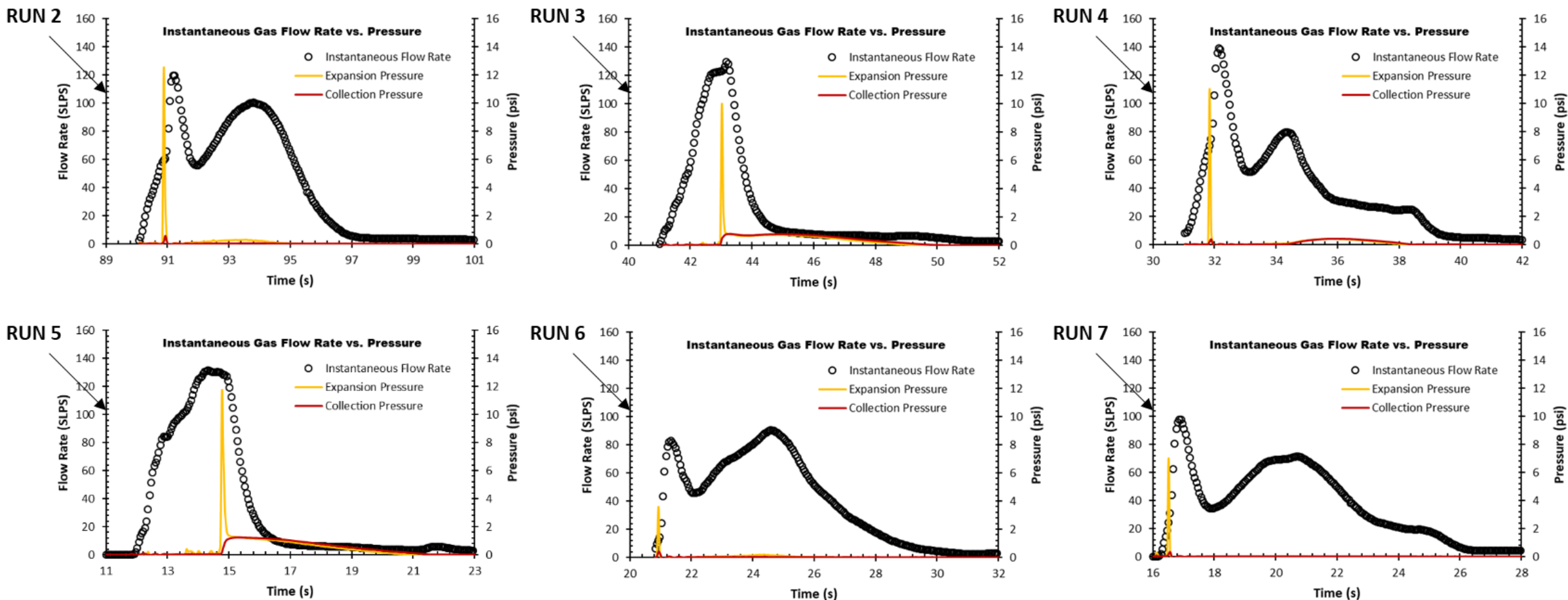
L-FTRC Results

Totalized Gas Volume vs. Totalized Air Volume



From: Rickman, S.L., et al., *Calorimetry for Large-format Lithium-ion Cell Thermal Runaway (TR), Volume II: GS Yuasa 134-Ah Cell Testing Summary*, NESC-RP-17-01291, April 30, 2020

L-FTRC Results



From: Rickman, S.L., et al., *Calorimetry for Large-format Lithium-ion Cell Thermal Runaway (TR), Volume II: GS Yuasa 134-Ah Cell Testing Summary*, NESC-RP-17-01291, April 30, 2020



Wrap-Up

Lithium-ion cells present the challenge of thermal runaway (TR).

Propagation of TR must be considered when designing lithium-ion batteries.

Design and analysis requires knowing not only total TR energy yield but also how the energy is distributed between that which conducts through the cell casing and that which is vented as gases and effluents.

Existing calorimetric techniques provide some but not all required data.

A new NASA-developed technique, called Fractional Thermal Runaway Calorimetry, provides data to ascertain total energy yield as well as distribution amongst various heat transfer modes.

FTRC allows rapid testing with fast turnaround enabling accumulation of statistically significant quantities of data.

Calorimeters have been developed and are in use for, both, small- and large-format cells.



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Thank You!

Questions?